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MEMORANDUM REPORT ARBRL-MR-02901

HMX, RDX, PETN, AND TNT REVISITED FOR SINGLE CRYSTAL AND VACUUM DROP WEIGHT SENSITIVITY

James E. Cole

February 1979

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
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HMX, RDX, PETN, and TNT were tested for impact sensitivity in a drop weight apparatus. The most common drop test found in the literature was the impact by drop weight onto multiple crystals of explosive set on sandpaper. We tested on sandpaper and then conducted additional tests on bare metal, with multiple and single crystals, and at ambient and in vacuum conditions.

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20. ABSTRACT: (Cont'd)

HMX and RDX were most extensively examined during the additional test to obtain baseline sensitivity controls for programs contemplated for either sensitizing or desensitizing the explosives. We found sandpaper tests to be too severe compared to the bare tool tests which gave us a better explosion point sensitivity spread.

Single crystals and vacuum tests consumed too much time without being more definitive. However, these tests did show that the absence of air to form hot spots had little effect on the compound's sensitivity.

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LIST OF FIGURES

I. INTRODUCTION

At the Ballistic Research Laboratory (BRL), the drop weight apparatus is used to provide supplemental data for explosive initiation sensitivity. These sensitivity data are for compliance with safety requirements prior to preparing explosive and propellant charges. The sensitivity data are also useful for estimating hazards by extrapolation.

The literature values for drop weight impact sensitivity of RDX and HMX were contradictory. These explosives with sandpaper on the anvil showed equal sensitivity, however, on a bare anvil HMX was more sensitive than RDX.[1,2] We needed to know which observation was correct before processing these explosives as dry powders.

Processing operations such as grinding and pressing fracture the crystalline explosive. Fracturing of crystals is not likely to cause reaction, even in lead azide which explodes only after compressional heating.[3] Experience has shown that pressure applied slowly in order to compact secondary explosives is safe. However, as a safety requirement, it is necessary to know that the crushing action will not cause initiation during compaction. If air is present in the chamber, ram clearances are sufficient to allow most of the air to move away from the compacted mass. Frictional heat from the moving crystals is transferred to the confining metal surfaces. During compaction, overheating is the main hazard and the drop weight impact apparatus causes an effect which can be considered predominantly thermal. To use the impact apparatus to study explosive friction and heating without the presence of air while maintaining crystal contact with confining metal surfaces required a new technique.

This report discusses the use of single crystals and vacuum techniques during drop weight testing on bare tool to determine the height for 50 percent chance of an explosion (H_{50}). Single crystals were used to eliminate the effect of compressed air associated with multiple crystal tests. Vacuum was used to eliminate follow-up compressibility of atmospherically tested single crystals and compressibility of air in multiple crystal samples. The net effect of using single crystal and vacuum is to allow the results to be interpreted as frictional reaction centers causing internal heating, i.e., hot spots without the influence of air.

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1. *NAVORD Report 4236, March 1956.*
2. *J. T. Rogers, "Physical and Chemical Properties of RDX and HMX," Holston Control No. 20-P-26, August 1962 (AD904410L).*
3. *M. M. Chaudhri, Combustion and Flame, 19, pp. 419-425 (1972).*

II. EXPERIMENTS

The explosives chosen for both the multiple crystals and single crystal tests were obtained through Army supply channels. Multiple crystals were screened through a number 50 sieve (297 μm opening) onto a number 100 sieve (149 μm opening). The screened sample was divided into a portion for the multiple crystal test and a portion for the growing of single crystals. To provide a purity record and attendant thermal history, differential thermal analysis (DTA) thermograms were obtained for each explosive.

The single crystals were grown from saturated solutions by evaporation of the acetone. Crystals of 2,4,6-trinitrotoluene (TNT) and pentaerythritol tetranitrate (PETN) had the regular crystal habit when grown without purification of the starting material; however, cyclotrimethylene trinitramine (RDX) and cyclotetramethylene tetranitramine (HMX) had the regular crystal habit only from certain containers used to grow crystals. These containers were made productive for growing crystals by washing with acetone and by the selective removal of crystal agglomerates during the first two evaporations.

The term "single crystal" is used here to describe a single, clear mass with sharp angles and plane faces. When the crystals were estimated to be at the correct weight, they were removed from the solution and dried between filter paper. After the crystals were dry, they were placed under vacuum, weighed, and grouped according to size. Sets of twenty single crystals and twenty multiple crystals were weighed into individual containers. All sets were kept in a desiccator until the impact tests.

The drop weight (impact) apparatus is bolted to a concrete pillar. The impact area is surrounded by an aluminum-framed, plastic box to protect the operator. We used the Type 12 tools shown in Figure 1.

The Type 12 components are listed below:

1. Anvil made from Ketos Steel, 59-60 Rockwell C.
2. Anvil support made from mild steel and bolted to base.
3. Hammer guide made from mild steel.
4. Hammer made from Ketos Steel, 59-60 Rockwell C.
5. Replaceable end for drop weight.
6. Drop weight-2kg size.

The tests were conducted at 22°C and a relative humidity of 50 to 70 percent. The anvil and hammer were changed after twenty drops or more often if their surfaces were excessively pitted. Sandpaper 5/0 coated with 180 grit garnet was positioned on the anvil for sandpaper tests.

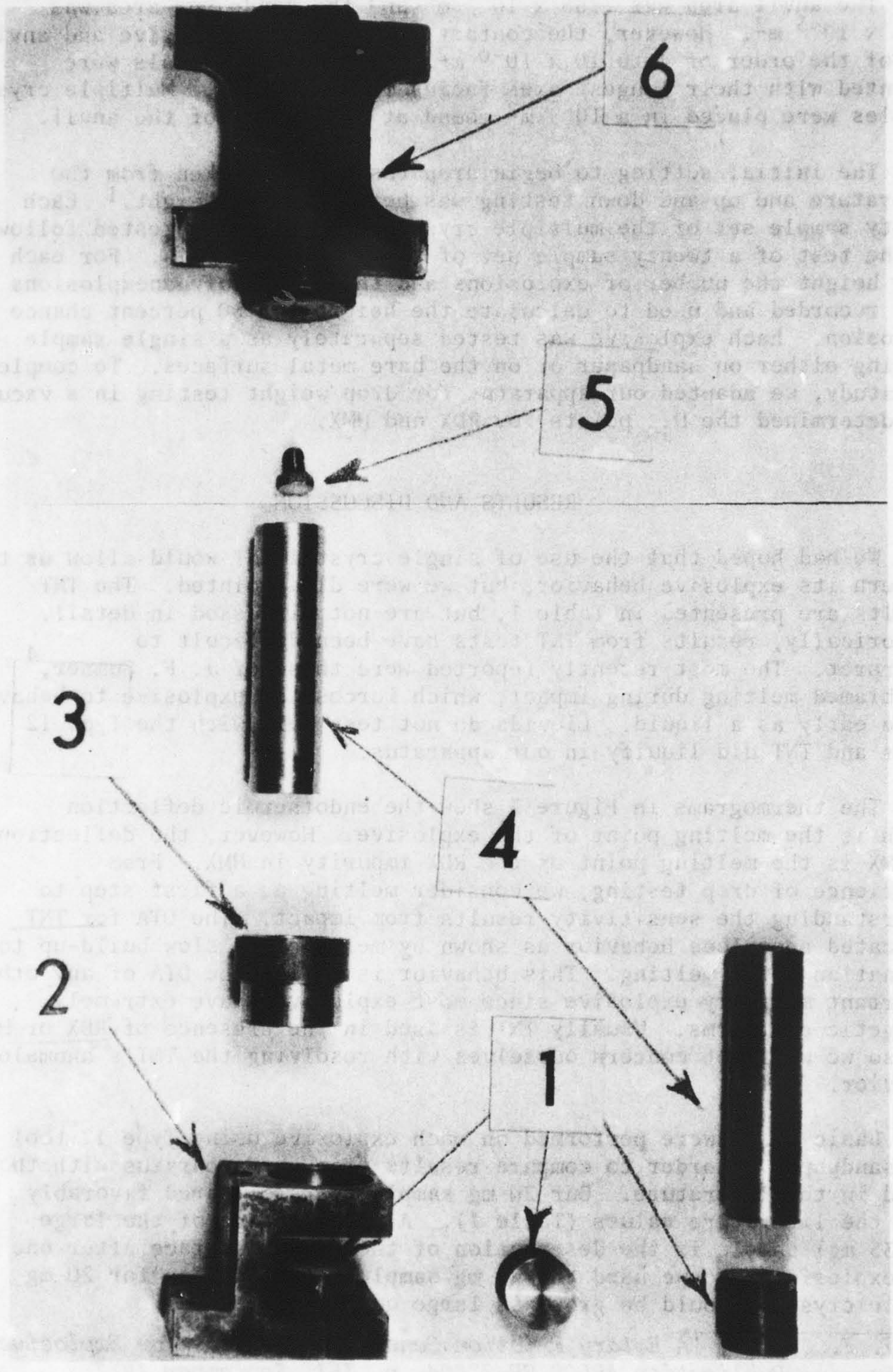


Figure 1. Type 12 Tool Components

The anvil area was $.755 \times 10^{-3}$ m^2 and the sandpaper area was $.645 \times 10^{-3}$ m^2. However, the contact area between explosive and anvil was of the order of 4 to 10×10^{-6} m^2. The single crystals were oriented with their longest axes facing front to back. Multiple crystal samples were placed in a 10^{-5} m^2 mound at the center of the anvil.

The initial setting to begin drop testing was taken from the literature and up-and-down testing was begun at that height.[1] Each twenty sample set of the multiple crystal explosive was tested followed by the test of a twenty sample set of the single crystals. For each drop height the number of explosions and the number of nonexplosions were recorded and used to calculate the height for 50 percent chance of explosion. Each explosive was tested separately as a single sample resting either on sandpaper or on the bare metal surfaces. To complete the study, we adapted our apparatus for drop weight testing in a vacuum and determined the H_{50} points for RDX and HMX.

RESULTS AND DISCUSSION

We had hoped that the use of single crystal TNT would allow us to discern its explosive behavior, but we were disappointed. The TNT results are presented in Table I, but are not discussed in detail. Historically, results from TNT tests have been difficult to interpret. The most recently reported were those of J. F. Sumner,[4] who blamed melting during impact, which forces the explosive to behave quite early as a liquid. Liquids do not test well with the Type 12 tools and TNT did liquify in our apparatus.

The thermograms in Figure 3 show the endothermic deflection which is the melting point of the explosive. However, the deflection in HMX is the melting point of the RDX impurity in HMX. From experience of drop testing, we consider melting as a first step to understanding the sensitivity results from impact. The DTA for TNT indicated anomalous behavior as shown by melting and slow build-up to detonation after melting. This behavior is unlike the DTA of any other important military explosive since most explosives have extremely energetic exotherms. Usually TNT is used in the presence of RDX or HMX and so we will not concern ourselves with resolving the TNT's anomalous behavior.

Basic tests were performed on each explosive using Type 12 tool and sandpaper in order to compare results from our apparatus with those found in the literature. Our 20 mg sample tests compared favorably with the literature values (Table I). A disadvantage of the large (30-35 mg) sample is the destruction of the tool's surface after one or two explosions, so we used the 20 mg sample. The samples for 20 mg single crystals could be grown in large quantities.

4. *J. F. Sumner, "A Rotary Friction Sensitiveness Test for Explosives," Special Publication ARLCD SP-77004, p. 351, September 1977.*

Table I. Impact (H_{50}) on Sandpaper Using 2kg Weight*

Material	Sample Wt. mg	H_{50}, cm	Material	Sample Wt. mg	H_{50}, cm
PETN			TNT		
M**	10	9	M	10	18
M	20	10	M	20	25
			M	30	51
S***	10	7	M	35	98
S	20	10	M	45	112
RDX			S	10	81
M	10	27	S	20	56
M	20	30			
S	10	10			
S	20	18			
S	30	20			
HMX					
M	10	30			
M	20	20			
S	10	13			
S	20	16			
S	30	23			

*Literature values[1] using a 2.5 kg weight and a 35 mg sample are as follows: PETN 10 cm; RDX 16 cm; HMX 16 cm; TNT 80 cm.

**Multiple crystal.

***Single crystal.

DTA THERMOGRAMS

Figure 2.

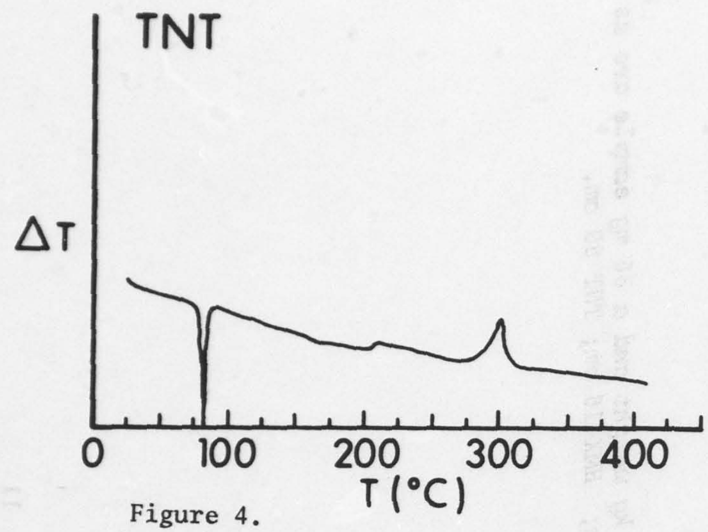


Figure 3.

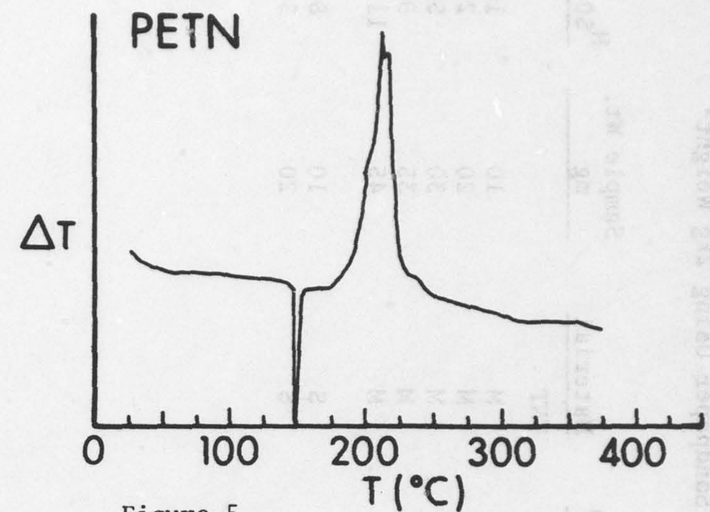


Figure 4.

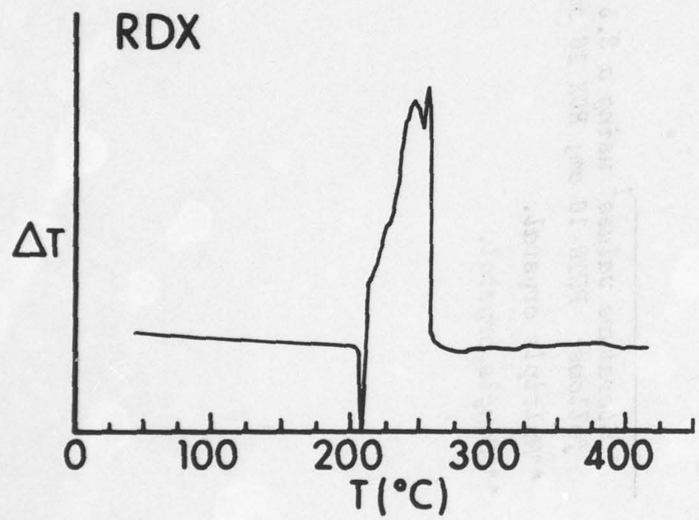
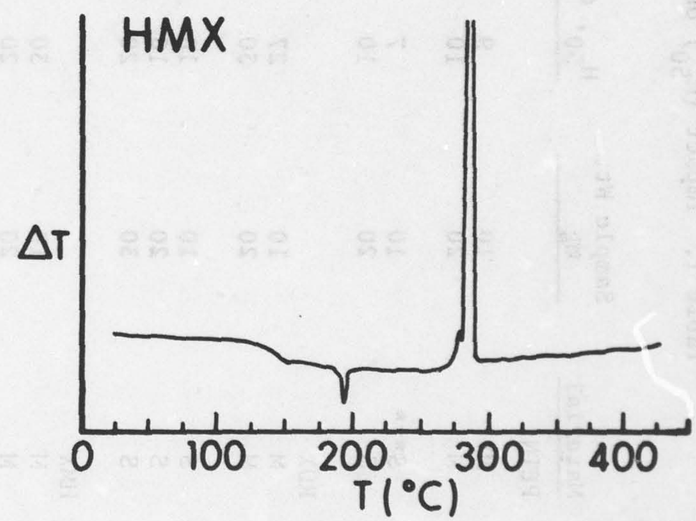


Figure 5.



Single crystal and multiple crystal tests compare H_{50} without and with the presence of air. For the 20 mg weight we found the single crystals of HMX and RDX to be much more sensitive than their multiple crystal counterpart. For PETN, the single crystal and multiple crystal were essentially the same for the 20 mg sample. We discontinued the testing on sandpaper because the grit and the sandpaper are not commonly encountered contact materials for an explosive.

The remainder of the tests were made with bare metal Type 12 tool surfaces. The bare metal provides a rapid heat transfer situation for the full contact of two metal surfaces instead of the metal and sandpaper surfaces. The H_{50} values increased.[4] The multiple crystal explosives changed sensitivity as recorded in Table II. Each multiple crystal explosive had a lower sensitivity as the H_{50} of PETN changed from 10 to 15 cm; RDX from 30 to 46 cm; HMX from 20 to 33 cm and TNT from 25 to 102 cm. The change can be attributed to the contact surfaces taking heat away rapidly. Therefore heating is different after melting and before explosion. To explode, heat must buildup as shown in the DTA curves.

Single crystal explosives that crush experience unequal heating by lateral movement during crushing. PETN is brittle; thus, the heat buildup is delayed by lateral movement. The particles are crushed, laterally scattered, and the H_{50} changes from 15 to 33 cm. The explanation seems to be that heat cannot rapidly buildup during the elapsed time before explosion. TNT is soft, melts during crushing, and has minimum lateral scatter. This results in higher heat concentration in the sample and the H_{50} changes from 102 to 53 cm. TNT is still an insensitive explosive. RDX and HMX are between soft and brittle. They scatter with even heating. Single and multiple crystal H_{50} values are nearly identifical with values of 43 and 46 cm, respectively for RDX and 33 cm each for HMX. Probably the minimum buildup of required heat for explosion occurs and the sample explodes. We would expect this from the DTA curves of these two explosives which rapidly proceed to explosion after melting. Only HMX is autocatalytic.[5]

To understand the mechanism further, we placed an inclosure around the apparatus and evacuated the interstitial air around the sample to prevent air compression. We did our testing in a vacuum of about 67 Pa. Testing in a vacuum made it very difficult to discern the noise of explosion from the noise of metal to metal impact. Smoke and spark were visible signs from outside the vacuum inclosure and so these were used as explosion indicators.

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5. R. N. Rogers, *"The Thermal Stability of HMX and RDX-Containing Systems," Naval Propellant Plant HMX Symposium, February 1962.*

Table II. Impact (H_{50}) Bare Tool Using 2 kg Weight*

Material	Sample Weight, kg	No Vacuum H_{50}, cm	Vacuum H_{50}, cm
PETN			
M**	20	15	
S***	20	33	
RDX			
M	20	46	41
S	20	43	46
HMX			
M	20	33	28
S	20	33	
TNT			
M	20	102	
S	20	53	

*Literature values[2] using a 5 kg weight and bare anvil are as follows: RDX 50 cm; HMX 32 cm.

**Multiple crystal.

***Single crystal.

The explosion in vacuum can be considered as the result of impacting a crystalline explosive subjected only to friction heating. The absence of air during crushing prevents compressive action and removes a source of hot spots and the cause of lateral scatter of crystals. Thus sensitivity of the H_{50} from only crystalline friction will be measured. The H_{50} impact explosion point in vacuum was determined for multiple crystals of RDX and HMX. The explosion points decreased 5 cm for each material indicating more severe conditions but the changes were insignificant to their relative sensitivity. We also checked the single crystal RDX in vacuum. The explosion point increased by 3 cm because the lateral movement slowed the build-up of heat. Again the sensitivity change is insignificant. This completed our study.

IV. CONCLUSIONS

To summarize, first the use of sandpaper on the anvil narrows the height range for all explosives subjected to the test. We omitted sandpaper and used a bare anvil. This spread the height range. Second, we investigated the absence of air in the test sample by impacting either a single crystal or a sample in a vacuum. Both techniques demonstrated that just crystalline friction is enough to cause the sample to explode on the Type 12 tool. Lastly, HMX is more sensitive than RDX. This was demonstrated in six separate tests:

1. The single crystal set on the sandpaper-covered anvil.
2. The multiple crystal set on the sandpaper-covered anvil.
3. The single crystal set on the bare anvil.
4. The multiple crystal set on the bare anvil.
5. The single crystal set on the bare anvil inclosed in a vacuum.
6. The multiple crystal set on the bare anvil inclosed in a vacuum.

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3. M. M. Chaudhri, Combustion and Flame, 19, pp. 419-425 (1972).

4. J. F. Sumner, "A Rotary Friction Sensitiveness Test for Explosives," Special Publication ARLCD SP-77004, p. 351, September 1977.

5. R. N. Rogers, "The Thermal Stability of HMX and RDX-Containing Systems," Naval Propellant Plant HMX Symposium, February 1962.

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